Lessons Learned:

Applying ISM to Experimental Work

at

Los Alamos National Laboratory

By

Jeff Williams

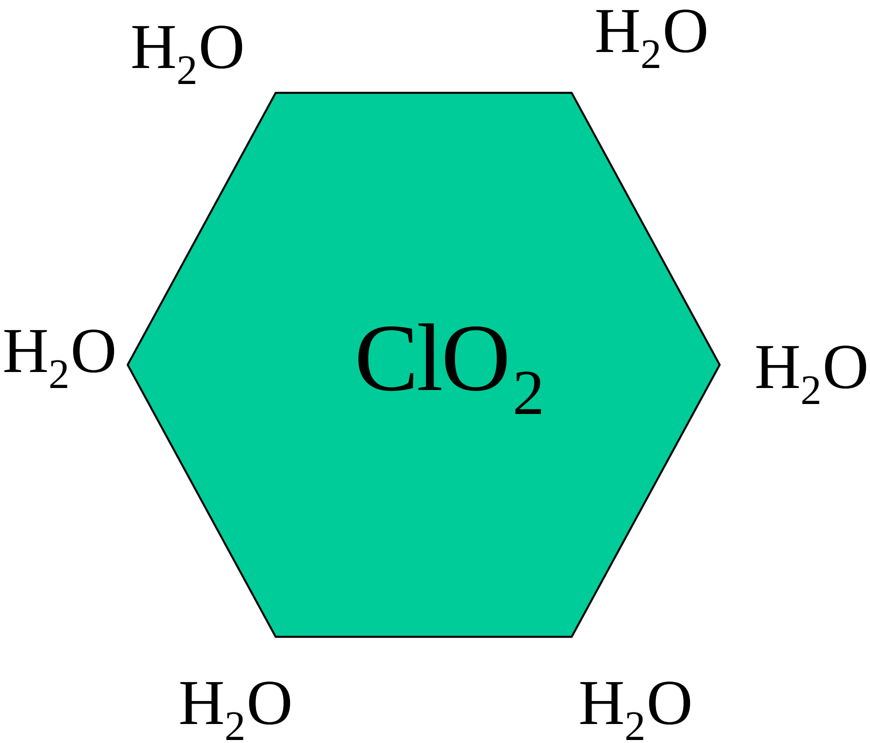
Facility Representative

NNSA Los Alamos Site Office

Reaction

$$Cl_2 + 2NaClO \rightarrow 2ClO_2 + 2NaCl$$

- **Objective of “Slurpee” was a high quality Hydrate Solid**
- **Prohibited DOT Explosive**
- **TNT equivalent of 0.46**
- **Hydrate form of ClO_2 is not dangerous**



What is ClO_2 Used for?

- **Bleaching:**
  - Flour
  - Paper
- **Biocide**
  - Kills Anthrax and other dangerous organisms.

Literature Research

- **Industry Generates ClO_2 by the metric ton at the point of use,**
  - Ensure concentrations stay below 10%
  - Use it immediately or store in water solution
- **Unstable with many possible initiators for decomposition**
  - Diffuse light
  - Shock
  - Heat
  - Impurities/intermediates

Similar Experimental Setup

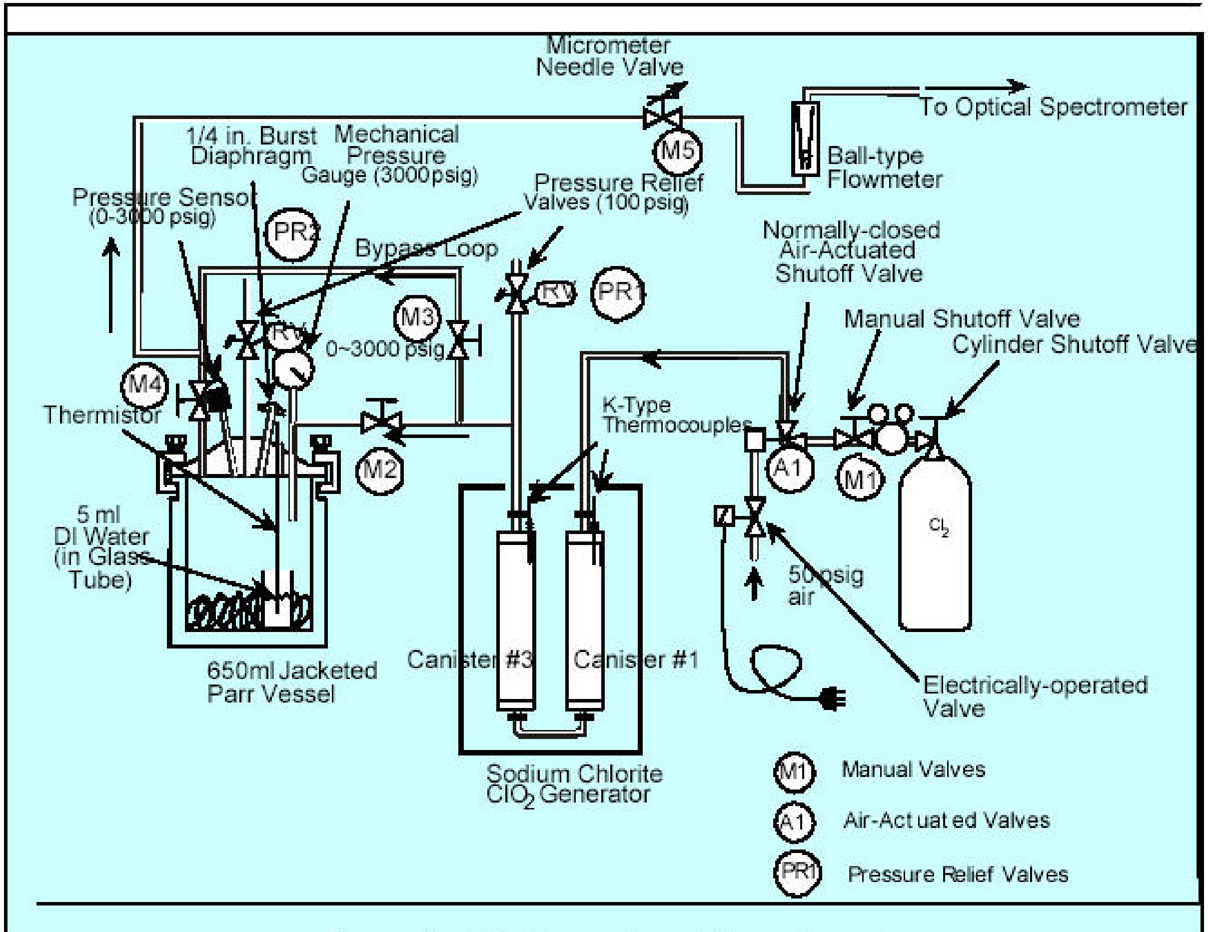


Figure 1 – Configuration of Experiment

Introduction

- **EVENT: Detonation of an estimated 68 grams of liquid (ClO_2) inside a Parr reaction vessel . The explosive force:**
  - turned the 3,000 psi. rated vessel into high velocity projectiles, penetrating a block wall
  - and generating a pressure front sufficient to destroy the fume hood
- **If the researchers had not noticed a temperature excursion, evacuated and been missed by all projectiles, it would have been a (really) bad day at work.**

Cartoon

An otherwise calm day at Los Alamos National Laboratory

As summarized
By
Gary Larson

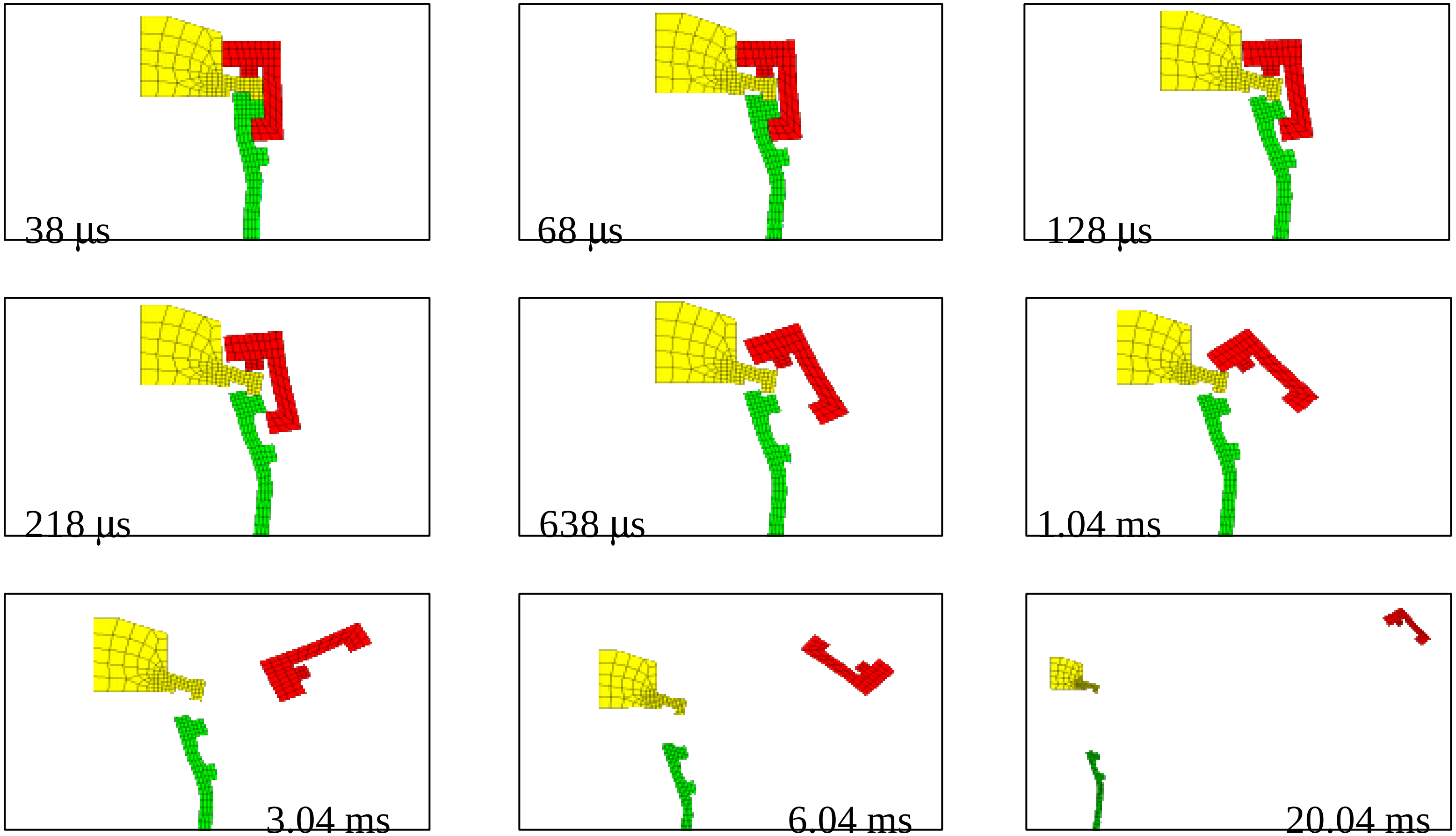
Action Sequence

- **The Day before:**
  - Experimenters introduce pure Chlorine and observe heat-of-formation spike, indicating likely saturation.
  - Shut off gas supply, maintain cooling and leave for the day.

Sequence Continued

- **The morning of:**
  - Experimenters replace Sodium Chlorite salt to ensure pure ClO_2 , avoiding impurities
  - Introduce Chlorine gas and verify with ten minutes of background data on spectrometer
  - Raise dip tube above water in vial
  - Divert flow into Parr vessel
  - Monitor generator and sample temperatures
  - Notice rapid temperature excursion at generator inlet
  - Evacuate and witness explosion

Finite Element Analysis



The analysis estimated the impulse to be **approximately 1900 psi-ms**

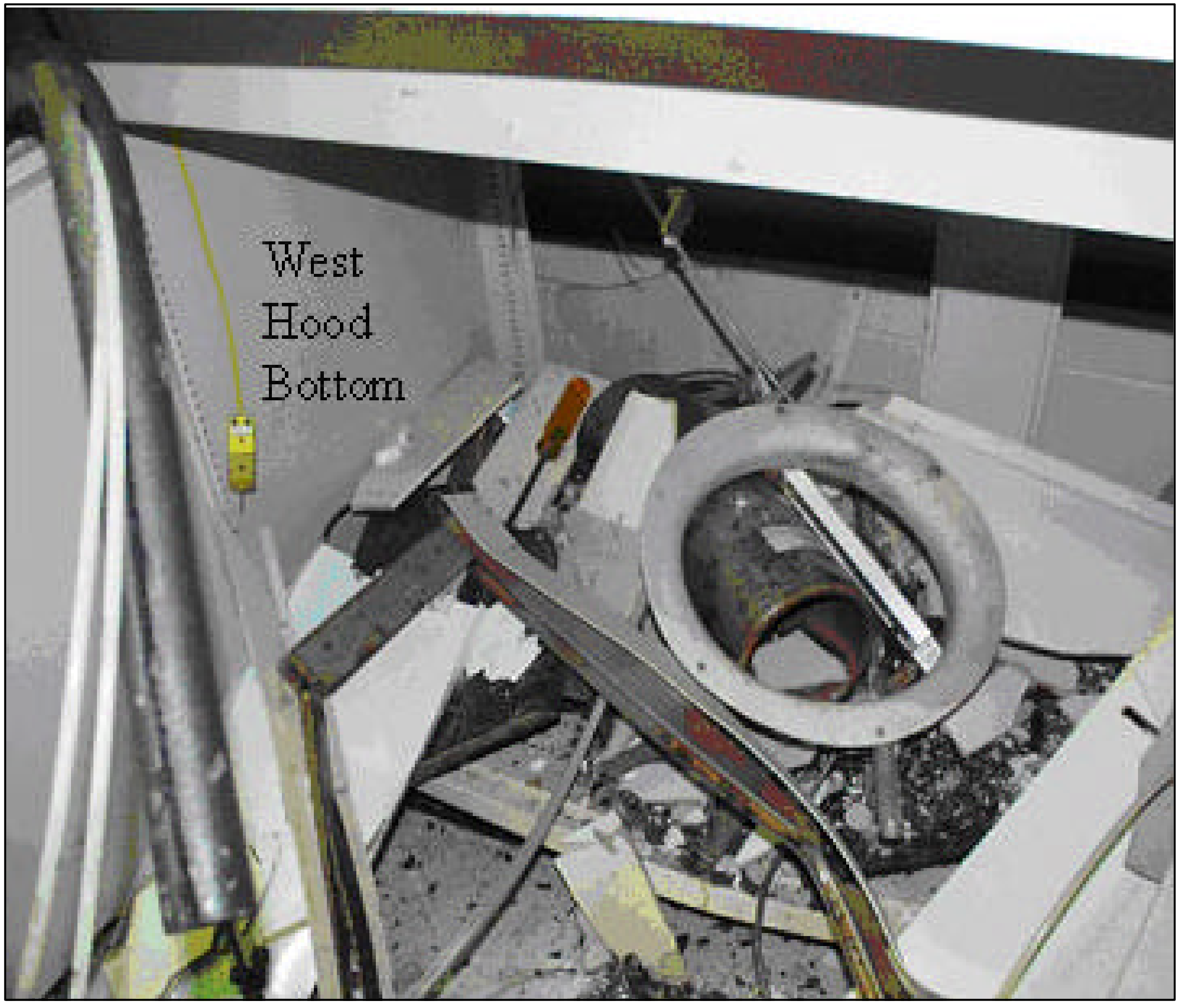
Hood After Event

Vessel Base / Witness

National Nuclear Security Administration

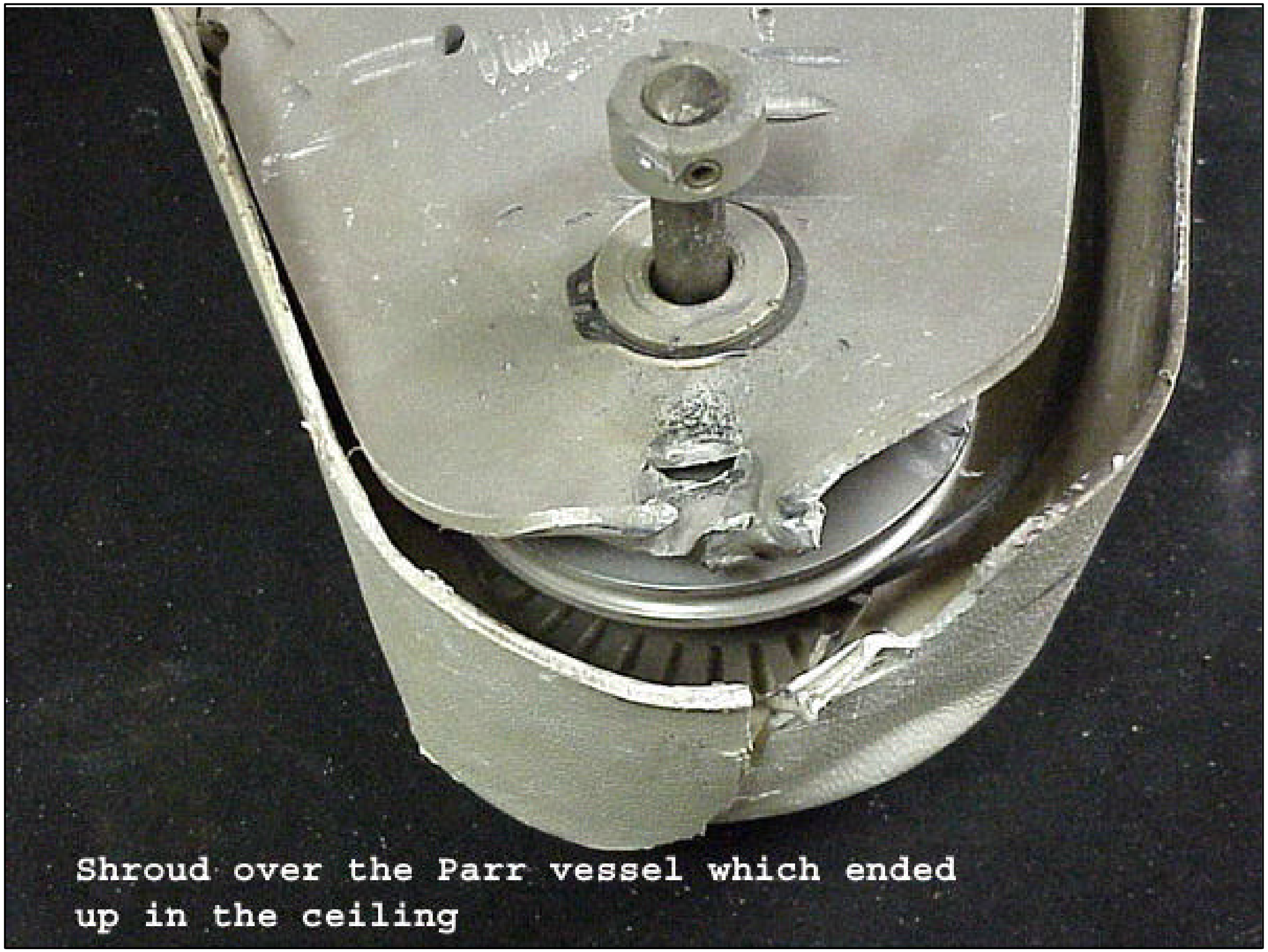
Department of Energy

Bottom of Hood

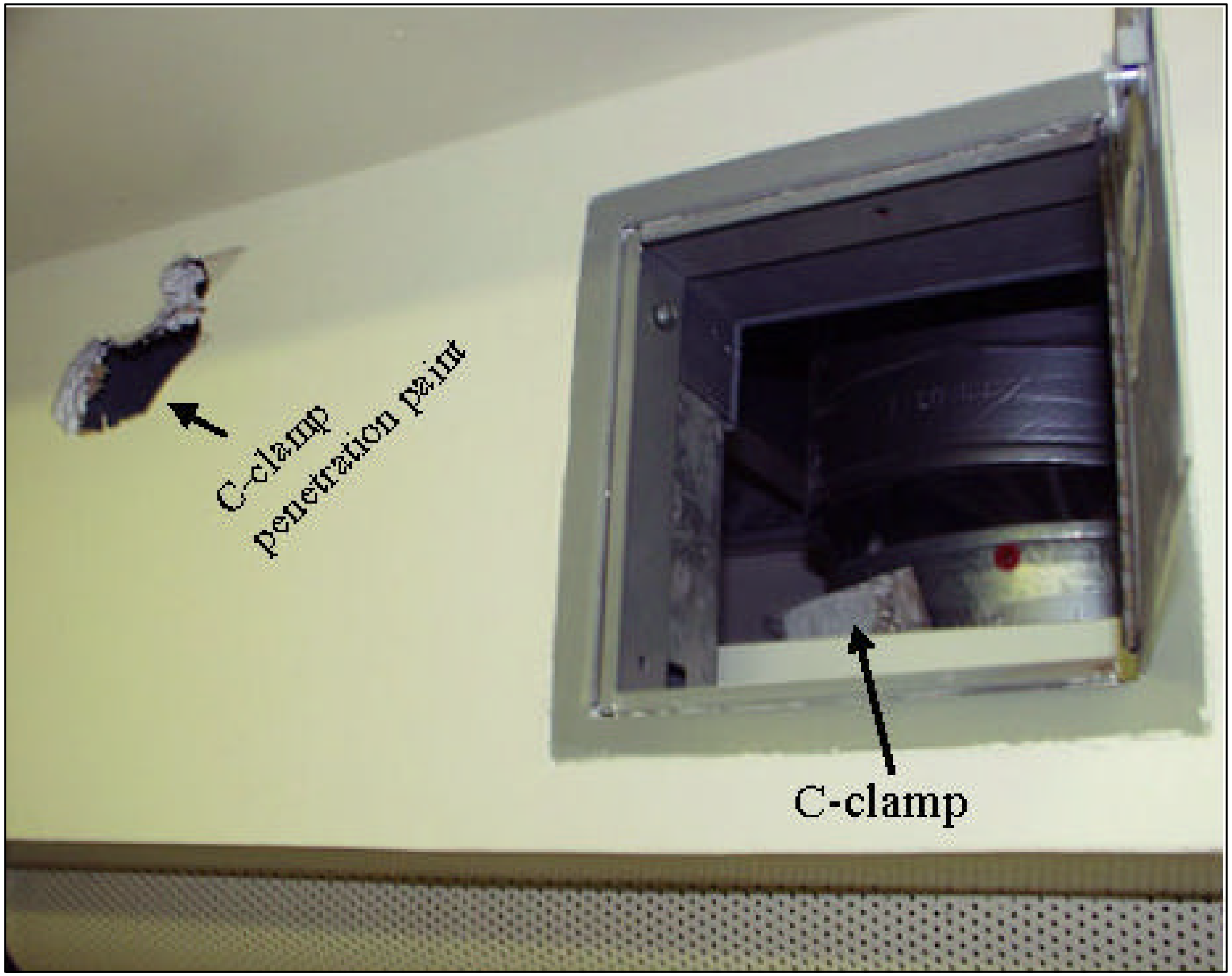


Ceiling Above Hood

Clamp Bolt Witness



Clamp Missile Witness



Analysis of Events Leading to the Explosion

- **Time Line Analysis**
  - Clarifies the steps that led this experiment outside the safety envelope.
  - Highlights the need for CLEAR definition of operational limits and approval by facility management.
- **Barrier Analysis**
  - Identifies several well intended, but inadequately analyzed safety assumptions.

Direct Cause

- **The Direct Cause of the accident was the failure of the researchers to recognize that liquid ClO_2 could form in the Parr vessel under the changed conditions of the experiment.**

Contributing Causes

- **The controls for handling pure chlorine were not appropriate and not properly placed.**
- **The system instrumentation was inadequate to detect the existence of conditions in the vessel that could lead to condensation of ClO_2 in to liquid.**

Contributing Causes cont.

- **The Parr vessel was not a suitable containment system for this experiment in that there was excessive confinement for a liquid phase explosion.**
- **The raising of the dip tube above the surface of the water in the vessel allowed ClO_2 to condense on the inner surface of the vessel and collect at the bottom, forming and explosive volume.**

Root Cause

- **The root cause of the accident was the performance of the "new" experiment utilizing 100% chlorine gas without a risk determination, formal hazard analysis, independent peer review, a revised HCP, and authorization to perform the work.**

Lessons for Energetic Materials Work

- **All dogs <u>can</u> bite you, (especially your own)**
  - Keep hazards and controls at the forefront of your thought process before and during work.
    - Use daily pre-work discussion
    - Post and discuss control checklists
    - Value pessimism – a vital element of science
  - Detail experimental controls, steps and precautions before beginning work.
    - Basis to objectively evaluate variances before making them, then document the safety basis as you would the experimental basis.

Lesson Two

- **A little can do a lot – this was done by 68 grams (less than 7 cu.in.) CLO_2**
  - Design experiments to balance cost, time, risk and amounts of energetics needed.
  - Use explosives, propellant and other criteria as guidelines.
  - Authorize work based on worst-case event.
  - This experiment only required 0.3 grams of CLO_2 to saturate the 5ml water sample, plus a small amount in the system.

Lesson Three

- **Quantify the unexpected, yet possible scenarios, then ensure controls preclude or mitigate them.**
  - Hazard Analysis identified formation of Liquid ClO_2 and that concentrations over 12% were unstable.
  - There was an administrative control of 4% Chlorine gas (8% CLO_2), and engineering control of low system pressure (>100 psig) far less than the phase change pressure of 258 psi.

Lesson Four

- **Utilize qualified SME's to evaluate, select and implement barriers & controls.**
  - The Parr Vessel for Slurpee was the primary engineered barrier to a deflagration, but was not certified for ANY pressure impulse, only static.
    - The calculated worst-case scenario was a gas phase deflagration, generating a pressure of 850 p.s.i
  - The (unidentified) hazard was actually a 75,000 psi reflected shockwave, resulting in a 1900 psi-ms impulse, due to confinement.

Lesson Five

- **Design and continually revisit your safety processes to ensure they are robust.**
  - The hazard screen identified explosives, but did not force an Explosives Safety Committee review
  - The screen also identified pressurized systems, but the criteria for formal review was 2 kilojoules stored <u>mechanical</u> energy, without regard to stored <u>chemical</u> energy.
  - The Safe Work Practice criteria identified the experiment as a low hazard, with minimal residual risk, thus no formal outside review was required.

Lesson Six

- **Safety Professional's proverb:**

"Dissertations do not mix well with procedures"

- **Keep safety documents as short, focused and quantified as possible.**
  - The HCP included adequate controls, but they were spread over 62 pages
  - Controls and experimental limits were not clearly identified and defined, did not specify caution levels, identify operating limits or shutdown conditions.
  - Controls were not independently reviewed by qualified engineers. (Safety, Mechanical, Instrumentation)

In the words of this experimenter…

- ***"Something just didn't feel right when we started the experiment that morning….."***

- ***"This was <u>really</u> dangerous, I could just feel it……."***

Credits

- Barbara Hargis, HSR-DO, Investigation Team Leader
- Scott Kinkead, DX-2, Explosive Chemistry
- Dave Bowman, DX-5, Pressure and Explosives
- Roger Kruse, PS-7, Investigator
- Mitch Harris, FWO, Facilities Expert
- Kirk Christiansen, Chair-Pressure Vessel Safety Committee